## (12) UK Patent Application (19) GB (11) 2 056 885 A

- (21) Application No 8025740
- (22) Date of filing 7 Aug 1980.
- (30) Priority data
- (31) 79/27601
- (32) 8 Aug 1979
- (33) United Kingdom (GB)
- (43) Application published 25 Mar 1981
- (51) INT CL<sup>3</sup> B05D 1/12 1/04 1/06
- (52) Domestic classification
  B2E 1103 1204 1205
  1300 1302 1323 1708
  1747 1748 402S 403S
  404S 423T 423U 424T
  424U 434T 434U 436T
  436U 438T 438U 443T
  443U 444T 448T 448U
  449T 449U 460T 460U
  473T 473U 474T 474U
  489T 489U 495T 495U
  501S 508T 508U 509T
  509U 511T 511U 520T
  520U BB
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- (58) Field of search
  B2E
- (71) Applicant
  Blundell-Permoglaze
  Limited
  Tyseley
  - Birmingham B11 2BD
- (72) Inventor

  Martin Louis Holliday
- (74) Agents
  Abel & Imray
  Northumberland House
  303–306 High Holborn
  London WC1V 7LH

## (54) Powder coating cellulose fibre substrates

(57) A cellulose fibre substrate such as a wood based material is coated by depositing a coating powder on it to form a coating and then using radiant heat to cure the coating powder. The coating powder typically contains a resin with a curing agent and, preferably, an adjuvant, such as a plasticiser or a wax, for lowering its softening temperature. The coating powder is preferably applied by an electrostatic method and the radiant heat is preferably applied in the form of infra-red radiation, typically having a wavelength in the region of from 1 to 5 microns.

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## **SPECIFICATION**

## Coating cellulose fibre substrates

5 This invention relates to the coating of substrates made from cellulose fibre materials such as, 5 for example, timber articles wooden boards, chipboard, hardboard, cardboard and paper. The high cost of solvents for conventional liquid coatings has made the use of powder coatings more attractive. However, although powder coatings have been used for some time for coating metal articles using electrostatically charged powder the attraction to the metal depends 10 on its electrical conductivity and it has been thought impossible to use the same techniques for 10 wood based substrates because they are substantially non-conducting. Another difficulty with the application of powder coatings on wood is that of applying heat to melt and cure the powder. For metal articles, this heat is usually supplied by gas, oil or electrically heated convection ovens, but this heat can affect a wood based substrate by causing moisture, sap or 15 resin to be exuded causing defects in the coating. Also changes in the physical dimensions of 15 the substrate can occur due to drying. It is an object of the present invention to provide a process for using powder coating for coating a cellulose fibre substrate. According to the present invention there is provided a method of coating a cellulose fibre 20 substrate including the steps of depositing a resin-containing coating powder onto the substrates 20 to form a coating thereon and subsequently using radiant heat to cure the coating on the substrate. In one especially preferred embodiment of the present invention, the coating powder is applied by an electrostatic charge process. In this respect, although the cellulose fibre substrates 25 have low electrical conductivity, it has been found that their conductivity is sufficient for a 25 coating powder as defined above to adhere to the surface, provided that either the substrate is of sufficient thickness to have adequate electrical conductivity for a single earthing point to be sufficient or the substrate is backed by an earthed conductive support, (which may be a sheet or non-continuous, such as a cross-rod conveyor) or has a plurality of earthing points distributed 30 over it. 30 The powder is advantageously applied by an electro static spray technique but other methods, such as fluidised bed techniques, may also be employed. A coating of powder can also be produced by allowing the powder to fall on to the substrate without using electrostatic attraction, for example by using a vibratory hopper. This is 35 particularly suitable for coating one surface of a substantially flat sheet, so that the powder is 35 held on the substrate by gravity. Alternatively, the powder may be applied by other methods such as fluidised bed techniques. The radiant heat may be applied by ultra-violet irradiation or electron beam irradiation but is preferably applied by infra-red irradiation from suitable lamps and it has been found that 40 wavelengths in the region from 1.0 to 5 microns provide the best curing of the powder coating 40 without overheating the substrate. Wavelengths in the range of from 1.0 to 1.5 microns are generally preferred but infra-red radiation having a wavelength in the region 1.5 to 5 microns can be used and produces good results. Intensities of the shorter wavelength infra-red radiation found suitable are about 2 to 10 watts per centimetre of lamp length and 1 to 5 watts per 45 centimetre of lamp length for the longer wavelength radiation. A suitable distance of the lamps 45 from the substrate is from 2 to 20 cm, a shorter distance than this having been found to cause scorching of the powder and a greater distance having been found to cause the substrate to overheat before the powder is fully cured. The term "cellulose fibre substrate" includes, for example, wood, timber, chipboard, particle 50 board, fibre board, hardboard, cardboard, plywood, veneer, block board (which board products 50 may be paper-faced) and paper, including articles made from regenerated cellulose. Such substrates may, as in certain commercially available materials, especially chipboard and particle board, be pretreated, eg by impregnation or coating with a resin such as a polyester, polyurethane or alkyd resin. However, in accordance with the present invention, the coating 55 powder is applied directly onto the substrate. That is to say, it is not necessary to apply a 55 protective layer of resinous, eg polyester resin, material to such compressed boards before the coating powder is applied. The invention is especially suitable for coating a substrate of any wood based material. Such material has a small electrical conductivity due to its water content which, it is believed, enables the substrate to provide, if required, adequate electrostatic 60 attraction for the powder. If the substrate is relatively thick then the electrical capacitance of the substrate has been found to be adequate to hold the electrostatically charged coating powder

whilst it is cured by radiant heat, using a single earthing point on the substrate. If, however, the

substrate is thin then it is preferable to provide an electrically conductive backing which is connected to earth or at least a plurality of distributed earth points so that the electrostatic

65 charge induced into the substrate by the coating powder can be discharged to earth.

	The powder may be applied by an electrostatic spray gun to the substrate using conventional powder spraying equipment such as that known by the names Volstatic, Gema and Controsion. Alternatively the powder may be placed in a vibratory hopper under which the substrate to be	
5	coated is passed, the hopper being maintained at a suitable electrical potential to provide the required electrostatic charge to the particles sufficient to give a thin even film of powder on the surface of the substrate, or by a pure electrostatic method, eg by using the Brennenstühl apparatus. Alternatively the powder may be placed in a vibratory hopper under which the substrate to be coated is passed, using gravity rather than electrostatic charging to apply an even film of powder. The substrate whilst being coated with powder using an electrically	5
10	charged spray may be hung vertically from a fixed point or a conveyor to provide an earthing point for the substrate. Several earthing points would be provided for large areas or thinner sheets so as to provide adequate electrical conductivity from the surface being coated to earth. Alternatively the substrates may be coated in a horizontal position using a flat conveyor passing the sheets under fixed or reciprocating electrostatic spray guns. The conveyor may have an	10
15	electrically conductive support for the substrate so as to provide a good earth connection over the back face of the material.  After the powder coating is formed on the substrate it is cured by subjecting the powder film to radiant heat, preferably in the form of infra-red radiation. It has been found that the	15
20	wavelength, intensity and separation of the lamps from the substrate need to be selected carefully to ensure a good flow of the coating during curing and to complete the cure of the coating without overheating the substrate. Infra-red lamps producing radiation in the wavelength range from 1.00 to 1.5 microns may be provided spaced from 2 to 20 cms from the substrate so as to provide a power intensity of from 2 to 10 watts per centimetre of lamp length and	20
25	preferably about 4.3 watts per centimetre. Alternatively the lamps may produce radiation of wavelength in the range 1.5 to 5 microns and spaced the same distance from the substrate as for the shorter radiation to produce a power intensity of from 1 to 5 watts per centimetre of lamp length and preferably about 2 watts per centimetre.	25
30	It has been found that using the coating compositions described below a period of between 45 seconds and 2 minutes is required for a white powder coating and from 20 seconds to 2 minutes, especially 30 seconds to 1½ minutes, for a black powder coating to effect curing of the coating.	30
35	Although, in principle, it has been found that many commercially available powder coating materials can be applied electrostatically to cellulose fibre substrates with varying degrees of success and can be caused to flow and cure by using applied radiation as described above it has been found that greatly superior coatings can be obtained using coating compositions which are specifically intended for the purpose.	35
40	Thus the preferred coating compositions used in the present invention comprise a thermosetting resin, together with suitable curing agent, and an adjuvant which reduces the softening temperature of the resin composition, for example a flow agent, a plasticizer, or, preferably a wax, or a combination of two or more such materials. Formulating the coating composition with the above-mentioned ingredients, it is possible, especially when electrostatic application and infra-red radiation curing methods are employed, to obtain surface coatings which exhibit a greatly enhanced appearance and surface hardness, as measured, for example, by using the	40
45	FIRA BS 3962 scratch test. This degree of surface hardness gives an excellent resistance to scuffing, coin marking and handling, but still provides the adhesion and flexibility necessary for sawing and drilling operations.	45
50	The thermo setting resin is preferably a polyester resin, but other resins may be employed. The resin used should preferably have a softening temperature between 70°C and 120°C, a Tg of between 40°C and 80°C, a melt index between 2 gm/min and 30 gm/min (ASTI D1238–62T) and a density of 1.1 to 1.4 gm/ml at 23°C. If the resin is a polyester it may incorporate esters of terephthalic and similar polyacids and polyols such as glycols with from 2 to 10 carbon atoms. The curing agents may be mainly functional through glycidyl groups, i.e. so as to produce a pure polyester, and may be trifunctional materials such as Triglycidyl	50
55	Isocyanurate (TGIC) and Tri(epoxy propyl) Isocyanurate (TEPIC). Other suitable curing agents include epoxy resins of epoxide equivalent between 400 and 1,000, and isocyanates, which produce polyurethanes.	55
60	Alternative coating powders may be based on other resins, e.g. epoxy resins, such as those cured with substituted or unsubstituted dicyandiamides, amines, amidines or anhydrides; acrylics; epoxy-phenolics; epoxy-novolacs and epoxy-resols; the curing systems may in some cases contain an accelerator.  In general the coating powder will incorporate a flow agent to reduce its surface viscosity. The flow agent is typically employed in a proportion known to those in the art, for example, in an	60
65	amount of at least 0.1 parts by weight based on 100 parts of the resin, and advantageously in an amount of from 0.2 to 10 parts, especially from 0.4 to 5 parts, although the exact amount employed will, of course, depend on the nature of the resin, the flow agent, and the other	65

	ingredients of the coating powder. Amongst suitable flow agents there may be mentioned, for example, polyacrylates, fluorocarbon polymers, silicone polymers, polyvinyl butyral polymers etc. The preferred coating powders incorporate a plasticiser or, more especially, a wax, to bring about a reduction in softening temperature.	
5		5
10	polyethylene and polypropylene waxes, amide waxes, e.g. bis-stearamide, gums and other high molecular weight materials, including crystalline and microcrystalline waxes.  Depending on the application concerned, the coating powders may also comprise various.	10
15	other ingredients, including, especially, pigments for coloured coatings and non-pigmenting fillers as extenders. Amongst suitable pigmenting fillers there may be mentioned, titanium dioxide, carbon black, iron oxides, lithopone, zinc oxide and pigments based on cadmium, lead or chromium, as well as organic pigments such as phthalocyanines, are and dioxazine dyestuffs.	15
20	etc.; amongst suitable extenders there may be mentioned, for example, calcium carbonate and barium sulphate, magnesium carbonate, silica, talc, silicates and other mineral fillers.  The pigments are preferably employed in amounts of from 0.5 to 100 parts by weight per 100 parts of resin, typically from 10 to 50 parts. It should be emphasized, however, that the present invention also provides clear finishes, in which case the pigment may be excluded. The	20
25	extenders, it present, are typically employed in amounts of up to 100 parts by weight per 100 parts of resin, especially from 5 to 50 parts, although amounts of up to 200 parts by weight may be used for so-called "flat" finishes.	25
	of the present invention is used to coat paper products.  In addition, the coating powders may include other ingredients which may be added, for any given purpose, including, for example, plasticizers, ultra violet absorbents, accelators, anti-cratering agents, catalysts and fungicides etc.	20
30	A typical white coating powder for use in the present invention comprises; 100 parts by weight resin, e.g. polyester 4-20 parts by weight curing agent, e.g. triglycidyl isocyanurate	30
35	0.2-2 parts by weight flow agent, e.g. polyacrylate 10-80 parts by weight pigment, e.g. titanium dioxide 0-60 parts by weight extender, e.g. calcium carbonate 0.1-10 parts by weight wax, e.g. polyethylene. A typical black coating powder for use in the present invention comprises;	35
40	100 parts by weight resin, e.g. epoxy, 2–10 parts by weight curing agent, e.g. amine 0.2–2 parts by weight flow agent, e.g. polyacrylate 0.1–0.6 parts by weight pigment, e.g. carbon black	40
45	0-60 parts by weight extender, e.g. calcium carbonate 0.1-10 parts by weight wax, e.g. polyethylene. The coating powders may be formulated by conventional techniques, for example, by premixing the components in a blender, extruding the blend and then grinding the extrudate; the preferred particle size being in the range of from 1 to 75 microns. The following Examples illustrate the invention, parts being by weight.	45
50	Example 1 The following samples illustrate coating powders which are suitable for use in the present invention.	50

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	A White Coatin	na Powder			
				Parts	
			Typical	Preferred Ranges	
5	Resin	Polyester	100	100	5
	Flow agent	Polyacrylate	1	0.4-1.6	
	Curing agent	TGIC	7	5–15	
	Pigment	Titanium Dioxide	48	20-80	
	Filler	Calcium Carbonate	43	0–60	4
10	Wax	Polyethylene	3	1–6	10
	B Black Coatin	na Powder			
	Resin	Polyester	100	100	
	Flow agent	Polyacrylate	1.2	0.6-1.8	
15	Curing agent	TEPIC	7.1	4–15	15
. •	Pigment	Carbon Black	2.1	0.75-6	
	Filler	Barium sulphate	37.5	0-60	
	Wax	Polypropylene	3.1	0.75-4.5	
20	C Clear Coatin	na Povedor			20
20		Polyester	100	100	2.0
	Resin		17.6	12-22	
	Curing agent	Blocked isocyanate	1.7	0.75-3.5	
	Flow agent	Flurocarbon Amidine	0.36	0.75-3.5 0-2.5	
2 5	Accelerator		2.3	0-2.5	25
25	Wax	Amide	2.3	0.1-3.0	20
		red Coating Powder	400	100	
	Resin	Ероху	100	100	
00	Flow agent	Polyacrylate	0.65	0.4–1.0	20
30	Curing agent	Amidine	7	5-10	30
	Filler	Calcium Carbonate	55 05	40-80	
	Pigments	(Titanium Dioxide	35	20–60	
		(Iron Oxides	4.8	2–20	
35	Wax	Polypropylene	2.3	1–6	35
	E Structured I	Finish Coating Powder			
	Resin	Epoxy Novolac	100	100	
	Flow agent	Polyacrylate	3.7	1.5-7.5	
40	Curing agent	Phenolic	43	35-50	40
	Pigment	Titanium Dioxide	61	50-100	
	Filler	Barytes	35	0-50	
	Wax	Polyethylene	1.5	1.0-4.0	
45	F Paper Coati	ng Powder			45
	Resin	Polyester	100	100	
	Flow agent	Silicone	1.15	0.75-2.0	
	Curing agent	TEPIC	7.15	6-15	
	Filler	Calcium Carbonate	3.2	0-60	•
50	Wax	Amide	2.9	0.5-6	50
•	G Chemical R	Resistant Coating Powde	er e		
	Resin	Ероху	100	100	
	Curing agent	Phenolic	33	25-40	
55	Flow agent	Polyvinyl Butyral	4.5	2-10	<b>55</b>
	Pigment	Titanium Dioxide	77	45-90	
	Accelerator	2-Methyl Imidazole	0.35	0.25-0.75	
	Filler	Dolomite	10.6	0-45	
	Wax	Polypropylene	1.8	1.0-4.5	
60		- 15 - 5 1	- · · <u>-</u>		60
	Example 2				
	•	nented coating as in Fxs	ample 1A w	as sprayed onto 6' 6" × 2'6" hardboard flush	
	doors. The powder was applied using 3 electrostatic Powder spray guns mounted vertically over				-
				onveyor. The powder was applied uniformly to	•
65				nickness) after an initial dusting process. The	65
	_	•		•	

5	coated door was conveyed under Infra Red lamps of 1 micron wavelength for a total time of 75 seconds, during which the powder fused and cured. Following a short air jet cooling section, the doors were off loaded and stacked. The resultant finish was smooth and very hard, and could be sawn and drilled without damage or loss of adhesion.	5
ŋ	Example 3	
10	A beige textured finish powder as in Example 1D was coated onto sheets of fine surface chipboard. The board was supported on a cross rod conveyor, and first passed through a dust removal process. The powder was applied via a vibrated trough, dropping powder vertically onto the board, to give a film weight of 70 gms/sq metre (55 microns film thickness). The powder was fused and cured by passing the board under Infra Red lamps of 2–3 micron wavelength for a total time of 60 seconds. After air jet cooling the boards were off loaded and stacked. The finish had an even, smooth texture, suitable for use in kitchen furniture or shelving.	10
15	Various modifications and developments falling within the scope of the present invention will be apparent to those skilled in the art.	15
20	1. A method of coating a cellulose fibre substrate including the steps of depositing a resincontaining coating powder onto the substrate to form a coating thereon and subsequently using radiant heat to cure the coating on the substrate.  2. A method as claimed in claim 1, wherein the substrate is electrically connected to earth during deposition of the coating powder, the powder being electrostatically charged and caused	20
25	to impinge on the substrate.  3. A method as claimed in claim 2, wherein the powder is projected towards the substrate by means of an electrostatically charging spray gun.  4. A method as claimed in claim 2, wherein the powder is arranged to fall on the substrate	25
30	from a vibratory hopper which is maintained at a suitable electrical potential.  5. A method as claimed in any one of claims 2 to 4, wherein the substrate is thin and is backed by an earthed electrically conducting support during deposition of the coating powder thereon	30
35	radiation.	35
	9. A method as claimed in claim 8, wherein the infra-red radiation has a wavelength in the range 1.0 to 5 microns.  10. A method as claimed in claim 8, wherein the wavelength of the radiation lies in the	40
40	range 1.0 to 1.5 microns.  11. A method as claimed in claim 9, wherein the intensity of the radiation incident on the powder is between 1 to 10 watts per centimetre of length of the radiation source.  12. A method as claimed in any one of claims 8 to 11, wherein the infra-red radiation is	40
45	provided by lamps spaced between 2 and 20 cms from the powder coating.	45
50	between 40°C and 80°C.  15. A method as claimed in any one of claims 1 to 14, wherein the resin has a melt index between 2 gm/min and 30 gm/min (ASTM D1238-62T).  16. A method as claimed in any one of claims 1 to 15, wherein the resin has a density of	50
55		55
60	and the curing agent is an isocyanate or an epoxy resin.  19. A method as claimed in any one of claims 1 to 16, wherein the resin is an epoxy resin and the curing agent is substituted dicyandiamide, an amine, an amidine or an anhydride.  20. A method as claimed in any one of claims 1 to 16, wherein the resin is an acrylic, an epoxy-phenolic, an epoxy-novolac or an epoxy-resol.  21. A method as claimed in any one of claims 1 to 20, wherein the coating powder	60
65	comprises a flow agent.  22. A method as claimed in claim 21 wherein the flow agent is a polyacrylate, fluorocarbon, a silicone polymer or a polyvinyl butyral polymer.	65
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	amount of at least 0.1 parts by weight based on 100 parts of the resin.  24. A method as claimed in claim 23, wherein the flow agent is present in an amount in the	
5	range of from 0.2 to 10 parts.  25. A method as claimed in claim 24, wherein the flow agent is present in an amount in the range of from 0.4 to 5 parts.	5
	26. A method as claimed in any one of claims 1 to 25, wherein the coating powder comprises an adjuvant for lowering the softening temperature of the resinous composition.	
10		10
	30. A method as claimed in claim 27, wherein the wax is a crystalline or microcrystalline wax.  31. A method as claimed in any one of claims 27 to 30, wherein the wax is present in an	
15	amount of up to 10 parts by weight based on 100 parts of the resin.  32. A method as claimed in any one of claims 27 to 31, wherein the wax is present in an amount of at least 0.1 parts by weight based on 100 parts of the resin.	15
	33. A method as claimed in claim 32, wherein the wax is present in an amount of from 0.5 to 10 parts by weight based on 100 parts of the resin.	
20	34. A method as claimed in claim 33, wherein the wax is present in an amount of from 1 to 5 parts by weight based on 100 parts of the resin. 35. A method as claimed in claim 26, wherein the adjuvant is a plasticiser.	20
	36. A method as claimed in any one of claims 1 to 35, wherein the coating powder comprises a pigment.  37. A method as claimed in claim 36, wherein the pigment is titanium dioxide.	
25	38. A method as claimed in claim 36, wherein the pigment is carbon black. 39. A method as claimed in any one of claims 36 to 38, wherein the pigment is present in	25
	an amount of from 0.5 to 200 parts by weight based on 100 parts of the resin.  40. A method as claimed in claim 39, wherein the pigment is present in an amount of from 10 to 50 parts by weight based on 100 parts of the resin.	00
30	41. A method as claimed in any one of claims 1 to 40, wherein the coating powder comprises an extender.  42. A method as claimed in claim 41, wherein the extender is calcium carbonate or barium	30
	sulphate. 43. A method as claimed in any one of claims 41 or 42, wherein the extender is present in	25
35	an amount of from 5 to 50 parts by weight, based on 100 parts of the resin.  44. A method as claimed in any one of claims 1 to 43, wherein the coating powder comprises an ultra-violet absorbent, an accelerator, an anti-cratering agent, a fungicide or a	35
40	mixture of any two or more of said adjuvants.  45. A method as claimed in claim 1, wherein the coating powder is as described in Example 1 herein.	40
40	46. A method as claimed in claim 1, carried out substantially as described in either of Examples 2 or 3 herein.	
45	47. A method of coating a wood-based board substrate, wherein a coating powder comprising 100 parts by weight of a thermosetting resin, a curing agent for the resin, at least 0.1 parts by weight of a flow agent and up to 10 parts by weight of a wax is electrostatically deposited on the surface of the substrate and is then cured by infrared irradiation.	45
	48. A cellulose fibre substrate which has been coated by a method as claimed in any one of claims 1 to 42.  49. A cellulose fibre substrate as claimed in claim 48, which is timber.	
50		50